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High Speed, Radiation Hard CMOS Pixel Sensors for Transmission Electron Microscopy

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Abstract

CMOS monolithic active pixel sensors are currently being established as the technology of choice for new generation digital imaging systems in Transmission Electron Microscopy (TEM). A careful sensor design that couples μm-level pixel pitches with high frame rate readout and radiation hardness to very high electron doses enables the fabrication of direct electron detectors that are quickly revolutionizing high-resolution TEM imaging in material science and molecular biology. This paper will review the principal characteristics of this novel technology and its advantages over conventional, optically-coupled cameras, and retrace the sensor development driven by the Transmission Electron Aberration-corrected Microscope (TEAM) project at the LBNL National Center for Electron Microscopy (NCEM), illustrating in particular the imaging capabilities enabled by single electron detection at high frame rate. Further, the presentation will report on the translation of the TEAM technology to a finer feature size process, resulting in a sensor with higher spatial resolution and superior radiation tolerance currently serving as the baseline for a commercial camera system.

Keywords: Monolithic active pixel sensors, Transmission Electron Microscopy

1. Introduction: CMOS pixel sensors as TEM detectors

The need for a new generation of fast, direct electron detectors is well established in Transmission Electron Microscopy (TEM). Material science applications requiring in-situ imaging of dynamic processes at the atomic scale can benefit from high frame rate detectors, while direct electron detection with high spatial resolution and quantum efficiency is crucial in low dose applications such as biological cryo-EM. Besides sensitivity and readout speed, an ideal direct detector for TEM needs to minimize the adverse effect on position resolution of electron multiple scattering in the sensor substrate, and to provide adequate radiation tolerance in order to withstand the incident electron beam over a reasonable lifetime.

Traditional direct detectors such as film or image plates have large area and high granularity, but are slow and do not allow dynamic imaging. Optically-coupled CCD-based cameras present intrinsic limitations in both point spread function (PSF) and detective quantum efficiency (DQE) due to the presence of the scintillator and to back-scattering from the fiber optics, where the optical coupling is necessary as CCDs

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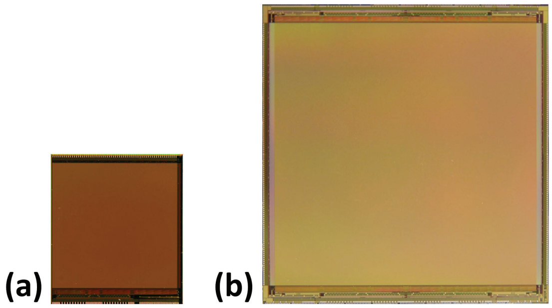


Fig. 1. Pictures of the (a) TEAM1k and (b) TEAM2k detectors, sized to scale for comparison. The two sensors feature respectively 1024×1024 and 2048×2048 square pixels of 9.5 μm pitch. TEAM2k occupies a full 21×23 mm^2 CMOS reticle.

are not radiation tolerant enough to be exposed directly to the electron beam. Moreover, they offer limited readout speed, typically not faster than video rate at 30 frames per second (fps).

CMOS monolithic active pixel sensors, first proposed in [1] for the detection of charged particles, have emerged over the past decade as the most attractive option for direct electron detection in TEM, as reported in several studies [2, 3, 4, 5, 6]. The low noise and integration capabilities offered by modern, deep-submicron CMOS manufacturing processes enable the fabrication of sensors with pixels of $O(\mu\text{m})$ pitch, exhibiting good signal-to-noise (S/N) performance for single electron detection, which greatly benefits DQE. As the sensitive volume is provided by the typically 5-15 μm thin epitaxial layer provided by the CMOS process, sensors can be back-thinned to thicknesses of ~50 μm and below to minimize the effect of electron back-scattering at the energies of interest to TEM, typically between 80 and 300 keV. The combination of small pixels and thin volume results in a superior PSF performance with respect to optically-coupled detectors up to large spatial frequencies. The use of CMOS technology opens new avenues for the sensor architecture: readout rates of several hundred fps are possible even for large area, megapixel scale sensors, and the availability of design techniques for radiation tolerant layout ensures a long device lifetime. Furthermore, complex processing capabilities can be integrated either on sensor or at the chip periphery, allowing the fabrication of compact camera systems with relatively low power dissipation.

2. The TEAM detectors

As part of the Transmission Electron Aberration-corrected Microscope (TEAM) project [7, 8] at the LBNL National Center for Electron Microscopy (NCEM), a detector R&D project led to the development of high frame rate, radiation-hard CMOS pixel sensors enabling 400 fps imaging with μm-level position resolution [9]. The sensors were manufactured in a commercial 0.35 μm CMOS process. Two chips have been fabricated, the 1024×1024 pixel TEAM1k and the 2048×2048 pixel TEAM2k, pictured in Fig. 1. The TEAM1k die size is approximately 10×11 mm^2, while TEAM2k occupies the full 21×23 mm^2 reticle offered by the manufacturing process. The full sensor design was performed using radiation tolerant layout rules [10]. Both sensors feature square pixels of 9.5 μm pitch, whose layout has been selected among several options from the evaluation of a small scale prototype in terms of electron detection capabilities and radiation hardness, as extensively detailed in [6, 11, 12]. Two versions of the TEAM1k die exist, an "imager" version that uses the full 1024×1024 pixel matrix as imaging area and a "diffraction" version in which the center of the imaging area features a 500 μm diameter diode used to collect the primary electron beam when the sensor is operated in diffraction mode. The diode both avoids severe damage to the imaging pixels caused by the ultra-bright beam, and serves as a monitor of the beam intensity, measured from the current through the reversely-biased junction. The sensors are back-thinned to 50 μm thickness and are mounted on proximity boards provided with a cut-out below the pixel matrix for the transmission of the electron beam and thus the minimization of back-scattering effects.

The TEAM1k chip architecture repeats sectors of 64×1024 pixels read out in parallel by 16 analog

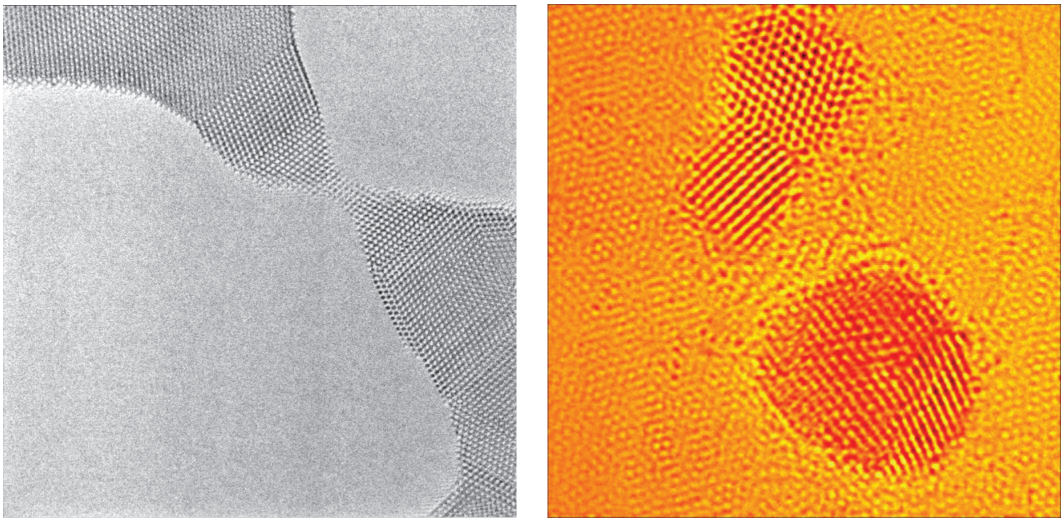


Fig. 2. Test images obtained with the 1024×1024 pixel TEAM1k sensor on the TEAM I microscope at the LBNL National Center for Electron Microscopy (NCEM). Left: Au nano-bridge image acquired with 300 keV electrons and 20 ms exposure time. Right: 80 keV electron image of AuFeO nanoparticles on a graphene layer, obtained from the average of 40 2.5 ms exposures. Images courtesy of P. Ercius, A. Gautam, C. Ophus, NCEM.

outputs. A pixel clock rate of 25 MHz ensures an integration time of 2.5 ms and enables operation at 400 fps in rolling shutter mode. Each output stage is provided with a low-power operational amplifier followed by a higher power slew-rate enhancement stage that is activated by large signals and ensure proper settling within the 40 ns pixel sampling period. TEAM2k uses the same basic blocks and scales up the architecture to 64 parallel outputs, with the option of multiplexing groups of 4 outputs to a single output to allow readout using the same DAQ system as for TEAM1k.

The TEAM1k sensor was commissioned in 2009, and a detailed characterization of its performance over an electron energy range of 80-300 keV has been reported in [9]. The sensor demonstrated unprecedented imaging capabilities at exposure times of 2.5 ms, up to three orders of magnitude faster than conventional, indirect detection techniques. PSF values from 12.1 μm at 80 keV to 7.4 μm at 300 keV were demonstrated in bright field imaging conditions (typical fluxes ≥5 e^-/pixel). The sensor is currently deployed on the sub-Å resolution TEAM I microscope at the LBNL NCEM. Sample images from experiments performed with the TEAM1k sensor are shown in Fig. 2. Tests of the diffraction sensor are currently in preparation, while TEAM2k is expected to be commissioned in 2012.

The TEAM1k sensor exhibited low pixel noise figures (~30 electrons) and an excellent S/N performance between 15 and 20 for single electron detection. This provided the basis for the demonstration of a new imaging technique applicable to low dose conditions. At fluxes <0.05 e^-/pixel, the signals generating from single electron interactions can be distinguished, and the electron impact positions can be reconstructed with larger accuracy with respect to the pixel pitch. High resolution images can thus be composed from the superimposition of several frames with sparse electron hits. The technique is called “cluster imaging” as it utilizes the spreading of the signal charge generated by a primary electron among groups of neighboring pixels (“clusters”) in order to improve position determination (e.g., by charge interpolation), a technique widely used in High-Energy Physics.

A simplified illustration of the concept is provided in Fig. 3, which reports experimental images of a Si[110] sample obtained on the TEAM I microscope with the TEAM1k sensor. A 512×512 pixel sub-matrix of the original images is shown in all panels for clarity. The image in panel (a), acquired with an electron flux of ~0.02 e^-/pixel and an exposure time of 2.5 ms, shows a sparse distribution of clusters generated by single electron events. Panel (b) shows the image obtained from simply superimposing 50 sparse frames acquired as in (a), while panel (c) shows a single bright field acquisition obtained with an electron flux of ~8 e^-/pixel and 2.5 ms exposure. The comparison of (b) and (c) shows that, even though the digital sum

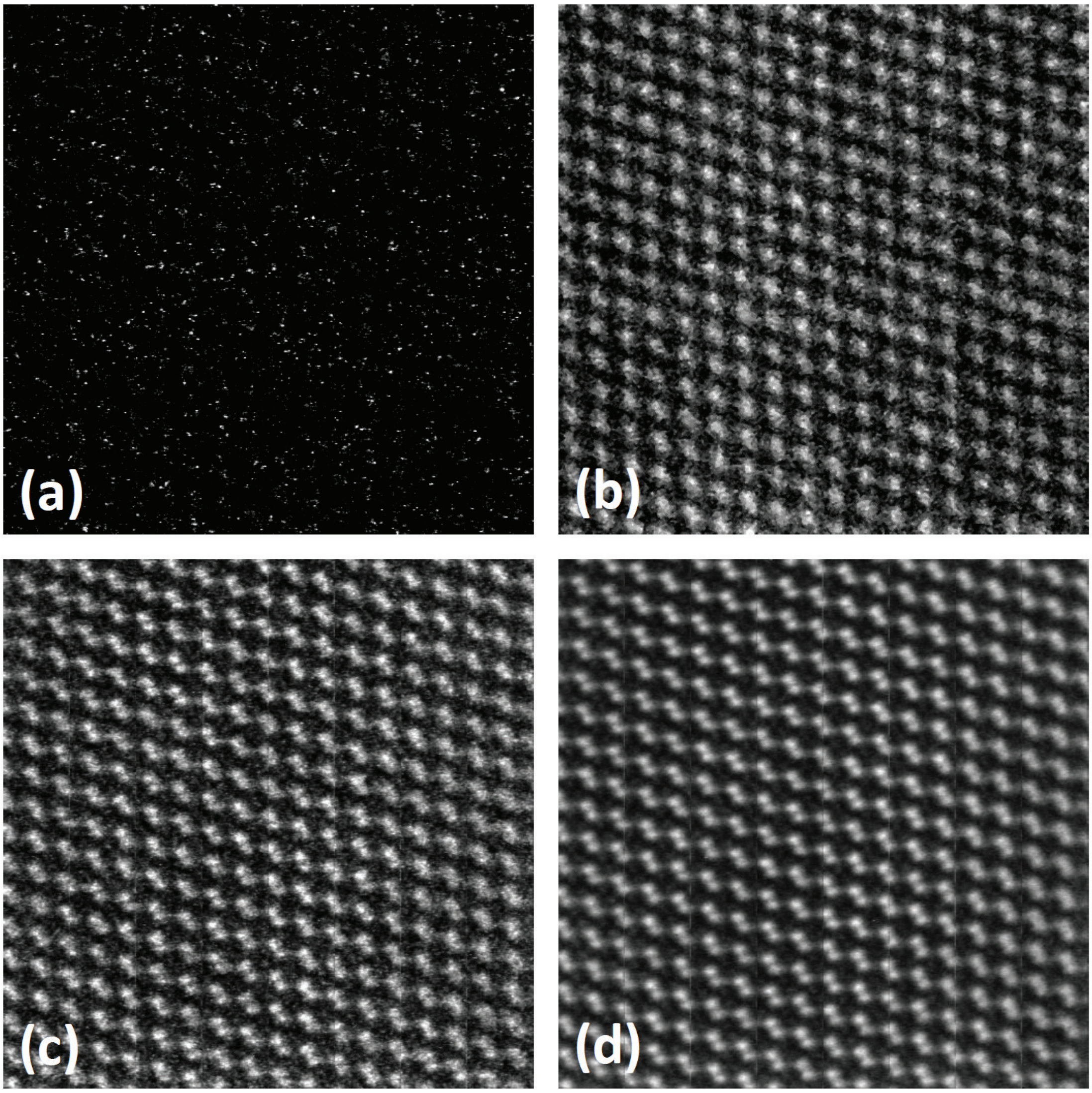


Fig. 3. Imaging of a Si[110] sample under different illumination conditions obtained with the TEAM1k sensor (512×512 pixel sub-matrix) and 300 keV electrons on the NCEM TEAM I microscope: (a) single 2.5 ms exposure with an incident flux of ~0.02 e^-/pixel; (b) digital sum of 50 2.5 ms exposures from the same dataset as in (a); (c) single 2.5 ms bright field exposure of the same sample as in (a) and (b) with an incident flux of ~8 e^-/pixel; (d) average of 10 bright field exposures from the same dataset as in (c). All images display raw, unprocessed data as acquired from the sensor.

of single electron images does not achieve the same resolution as the bright field image, an unambiguous image of the sample crystallographic structure becomes evident at an electron dose which is a factor 8 lower. Panel (d) shows the average of ten bright field exposures acquired as in (c), revealing a high-contrast image of the Si[110] "dumbbells" and their characteristic separation of 0.136 nm.

This qualitative illustration shows how high-frame rate, coupled with a high sensitivity sensor, may allow the acquisition of high resolution images at lower electron fluxes from the real-time reconstruction of single electron hit positions and their superimposition. Processing of the cluster charge distribution within the signal clusters using algorithms such as center of gravity or charge interpolation can further enhance the achievable resolution with respect to a simple binary discrimination. A resolution equal or better than in higher flux bright field conditions can thus be obtained at much lower electron doses, a capability which is crucial in applications such as biological cryo-EM where radiation damage to the sample is a major concern and the best possible resolution must be obtained at the lowest possible electron dose.

The cluster imaging technique was first demonstrated experimentally in [13] using data obtained on the TEAM1k sensor and employing a clustering algorithm based on S/N discrimination for cluster reconstruction, followed by charge interpolation for electron impact position determination. In this algorithm, the pixel matrix is first scanned to identify pixels whose S/N exceeds a certain preset threshold. This collection of "seed pixels" is then sorted and filtered to exclude pixels belonging to the same clusters from the list of candidate hits. The pixels surrounding each seed pixel in the filtered list are subsequently scanned against a secondary, lower S/N threshold for possible addition to the cluster. The electron impact position is finally calculated by center-of-mass interpolation of the signal charge in all the pixels thus included in the cluster. This analysis yielded a factor 2 to 3 improvement in PSF performance and ensured sub-pixel pitch position resolution throughout the whole electron energy range considered, from 6.7 μm at 80 keV and reaching 2.4 μm at 300 keV [9]. A variation of the same concept using a different analytical method was independently proposed in [14].

3. Second generation sensors in 0.18 μm CMOS process

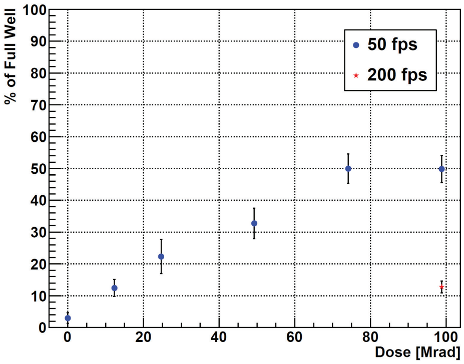
After the successful demonstration of the TEAM detector technology, the search for higher spatial resolution prompted us to evaluate finer feature size manufacturing processes, allowing smaller pixel size and intrinsically higher radiation tolerance and readout speed. A key aspect of this transition was ensuring that thinner epitaxial layers would still yield appreciable charge signal for single primary electron events, combined with low leakage current and noise to maintain adequate S/N performance. Further, special care was needed at the design level to ensure that dynamic range would be preserved under lower voltage operation. A smaller size of the charge-collecting diode in each pixel indeed translates directly into a smaller pixel capacitance and thus larger conversion gain, effectively reducing the usable full well for the same voltage swing.

Along the lines of the TEAM detector development, a small scale detector prototype was first designed in a commercial 0.18 μm CMOS process. The test sensor, funded by the Howard Hughes Medical Institute (HHMI) and referred to as HHMI in the following, implemented several pixel cell designs, arrayed on a 5 μm pitch and exploring various readout architectures, layout arrangements and processing options. The comparative test of the various designs led to the identification of an architecture with S/N performance comparable to the TEAM1k sensor and sub-pixel PSF for 300 keV electrons, improving from 3.9 μm in bright field conditions to 1.5 μm in cluster imaging [15]. The comparison between the TEAM1k and the HHMI PSF measurements under the two imaging modes is summarized in Table 1.

Chip	**TEAM1k**	**HHMI**
Pixel pitch	9.5 μm	5 μm
Bright field	(7.4±0.6) μm	(3.9±0.4) μm
Cluster imaging	(3.9±0.4) μm	(1.5±0.3) μm

Table 1. Comparison of Point Spread Function measurements performed on the TEAM1k sensor (0.35 μm CMOS process) and on the HHMI test sensor (0.18 μm CMOS process), for bright field and cluster imaging illumination conditions. All data refer to 50 μm thin sensors. Data compiled from [9] and [15].

A preliminary characterization of frequency-resolved Modulation Transfer Function (MTF) and Detective Quantum Efficiency (DQE) has been performed on the HHMI sensor with 300 keV electrons. At a spatial frequency corresponding to one-half of the Nyquist frequency, the MTF was measured to improve from 0.33 in bright field to 0.75 in cluster imaging, with corresponding DQE values of 0.3 and 0.5. The comparison of these figures with what achievable on standard, optically-coupled CCD camera systems shows an improvement of 2 to 3 times in bright field conditions, and up to 5 times with cluster imaging for both MTF and DQE.



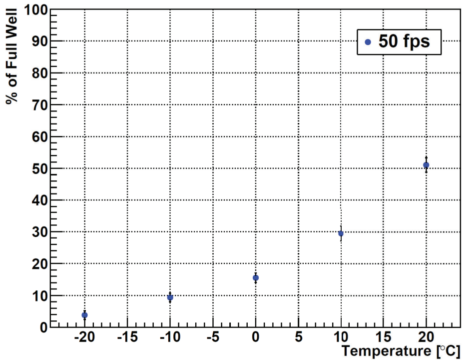


Fig. 4. Left: pixel leakage current, expressed as fraction of the sensor full well, as measured on the HHMI sensor as a function of the 300 keV electron dose. At the highest dose, the measurement was repeated by operating the sensor at a four time faster clock frequency (i.e., four times higher frame rate). Right: leakage current as a function of the cooling temperature as measured after the irradiation experiment, with the HHMI sensor operated at 50 fps. Data adapted from [15].

As expected from the scaling down of the minimum feature size, a significant performance improvement on the 0.18 μm process with respect to the 0.35 μm one used for the TEAM sensors was observed in terms of radiation hardness. The main effect of ionizing radiation on CMOS sensors is the trapping of positive charge in the field oxide, which causes inversion of the silicon at the interface and the generation of surface leakage current that flows into the charge collecting diode. This increase in leakage current can be corrected by dark level subtraction or by Correlated Double Sampling (CDS), but limits the usable dynamic range. Another consequence of leakage current is the increase of the shot noise contribution to the pixel noise. It is difficult to establish a typical dose requirement for TEM applications, as it is largely dependent on the actual usage and on the imaging mode. A rough estimate yields electron doses of the order of tens if not hundreds of Mrad for typical yearly usage, where the dose can increase dramatically in localized bright spots when the sensor is used for diffraction imaging. The radiation tolerance requirements for TEM detectors therefore become easily comparable with those in High-Energy Physics experiments [12].

The HHMI sensor radiation hardness was qualified with 300 keV electrons up to doses approaching 100 Mrad as detailed in [15]. At equal doses, a factor ~4 lower leakage current was measured on the HHMI sensor compared to the TEAM1k sensor. The relative effect on the usable dynamic range was however larger in the HHMI case due to the lower operating voltage of the 0.18 μm process with respect to the 0.35 μm one (1.8 V vs. 3.3 V). Figure 4(left) shows the increase in leakage current as measured on the HHMI sensor as a function of the 300 keV electron dose. The leakage current is expressed as the fraction of the sensor full well, showing that the dynamic range is effectively halved after the highest dose when the sensor is operated with a 20 ms exposure time, i.e. 50 fps readout. However, the data point at 200 fps included at the highest electron dose shows that a 4 times reduction in exposure time effectively reduces the effect of leakage current on dynamic range. At the same time, leakage current is strongly dependent on temperature and can thus be contained by proper cooling as shown in Fig. 4(right), where the pre-irradiation dynamic range is recovered by cooling the sensor to -20°C.

The HHMI pixel design was used in the development of the K2 sensor, a 16 megapixel, reticle size imager manufactured in the same 0.18 μm CMOS process, operated at 400 fps and thus streaming 6.4 gigapixels/s. A number of key design issues addressed in the scaling of the imager architecture from small scale prototypes to large area devices are described in [16]. The K2 chip has been designed and first commissioned at LBNL in 2011, and is currently being deployed in a commercial digital camera system, provided with processing capabilities for on-line cluster imaging integrated in hardware [17].

4. Conclusions

This paper has reviewed an LBNL-led effort that has successfully developed and deployed CMOS active pixel sensors as direct electron detectors for high resolution and fast imaging in Transmission Electron Microscopy. Direct detection on a thin sensor layer benefits both position resolution and quantum efficiency, while the use of advanced CMOS manufacturing processes enables the design of fast readout architectures which are at the same time very robust against radiation damage. Readout rates of several hundred frames per second have been demonstrated for large area, megapixel scale sensors. In particular, it has been shown that high resolution images can be obtained from single electron event processing at high frame rate and reduced electron doses. The translation of the 0.35 μm TEAM detector technology into a smaller feature size process (0.18 μm) ultimately resulted in sensors with μm-level position resolution and radiation hardness to very high electron doses, close to 100 Mrad. The 0.18 μm architecture is currently employed in a 16 megapixel, 400 fps commercial camera system.

Our R&D effort currently continues along the path of exploring finer feature size processes. The first prototype CMOS active pixel sensor manufactured in a 65 nm CMOS process and featuring pixels of 2.5 μm pitch is currently being tested, as introduced in [16]. An important advantage of very deep-submicron designs, aside from the improvement in resolution from the scaling of the pixel size and the superior radiation tolerance of thinner oxides, is the high level of integration possible, enabling for example the inclusion of on-chip integrated ADCs or signal processing units.

Acknowledgments

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